IS 7642 (1975): 4-Aminophenol [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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IS : 7642 - 1975

Indian Standard

SPECIFICATION FOR 4-AMINOPHENOL

UDC 667·21 : 547·564·4

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110001

[redacted] Gr 2

July 1975

AMENDMENT NO. 1 SEPTEMBER 1988
TO
IS : 7642 - 1975 SPECIFICATION FOR 4-AMINOPHENOL

[*Page* 4, *Table* 1, *col* 3, *Sl No.* (ii)] — Substitute '0·50' *for* '0·5'.

[*Page* 4, *Table* 1, *col* 3, *Sl No.* (iii)] — Substitute '0·50' *for* '0·5'.

[*Page* 4, *Table* 1, *col* 3, *Sl No.* (iv)] — Substitute '0·50' *for* '0·5'.

[Page 4, *Table* 1, *col* 3, *Sl No.* (v)] — Substitute '184·0 to 187·0°C' *for* '184 to 187°'.

[*Page* 4, *Table* 1, *col* 3, *Sl No.* (vi)] — Substitute '0·50' *for* '0·5'.

(PCDC 9)

Printed at Swatantra Bharat Press, Delhi, India

Indian Standard
SPECIFICATION FOR 4-AMINOPHENOL

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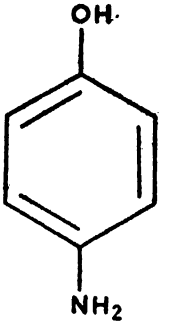
Indian Standard

SPECIFICATION FOR 4-AMINOPHENOL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 25 April 1975, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Chemical Division Council.

0.2 4-Aminophenol (C_6H_7ON), also described as 1-amino-4-hydroxybenzene, and *p*-hydroxyaniline, is an intermediate used for the manufacture of a large number of azo and sulphur dyes. It is represented by the following structural formula:



4-AMINOPHENOL
(MOLECULAR MASS 109·1)

0.3 This specification does not cover 4-aminophenol for use in pharmaceutical industry.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for 4-aminophenol.

*Rules for rounding off numerical values (*revised*).

2. REQUIREMENTS

2.1 Description — The material shall consist of white to light brown crystals. The material darkens on keeping, particularly on exposure to air.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR 4-AMINOPHENOL

Sl No.	Characteristic	Requirement	Method of Test, Ref to Cl No. in	
			Appendix A	IS : 5299-1969*
(1)	(2)	(3)	(4)	(5)
i)	Assay (excluding 2-aminophenol content), percent by mass, on dry basis, *Min*	98·0	—	12.1
ii)	Insolubles in hydrochloric acid, percent by mass, *Max*	0·5	A-1	—
iii)	Sulphated ash, percent by mass, *Max*	0·5	—	11.2
iv)	2-Aminophenol content, percent by mass, *Max*	0·5	A-2	—
v)	Melting point	184 to 187° with decomposition	—	8
vi)	Moisture, percent by mass, *Max*	0·5	—	9.3

*Methods of sampling and tests for dye intermediates.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in steel drums (*see* IS : 2552-1970*) lined with suitable polyethylene film, or as agreed to between the purchaser and the supplier.

3.2 Marking — Each container shall be securely closed and shall bear legibly and indelibly the following:

a) Name of the material;
b) Name of the manufacturer and his recognized trade-mark, if any;
c) Gross, net and tare mass;
d) Batch number; and
e) The word "POISON" in red, printed on white background [*see* Fig. 11 of IS : 1260 (Part I)-1973†].

*Specification for steel drums (galvanized and ungalvanized) (*first revision*).
†Pictorial markings for handling and labelling of goods: Part I Dangerous goods (*first revision*).

3.2.1 Each container shall, in addition, bear the minimum cautionary notice worded as under:

"KEEP WELL-CLOSED AND PROTECTED FROM LIGHT AND AIR. AVOID INHALATION AND CONTACT WITH SKIN."

3.2.2 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in **3** of IS : 5299-1969*.

4.2 Number of Tests

4.2.1 Test for assay shall be conducted on each of the individual samples.

4.2.2 Tests for the determination of all other characteristics given under Table 1 shall be conducted on the composite sample.

4.3 Criteria for Conformity

4.3.1 *For Individual Samples* — The lot shall be declared as conforming to the requirement of assay if each of the individual test results satisfies the relevant requirement given in Table 1.

4.3.2 *For Composite Samples* — For declaring the conformity of a lot to the requirements of all other characteristics tested on the composite sample, the test results for each of characteristics shall satisfy the relevant requirements given under **2** and Table 1.

5. TEST METHODS

5.1 Tests shall be carried out according to the methods prescribed in Appendix A and IS : 5299-1969* as indicated in col 4 and 5 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960†) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Methods of sampling and tests for dye intermediates.
†Specification for water, distilled quality (*revised*).

APPENDIX A

(*Table* 1, and *Clause* 5.1)

METHODS OF TEST FOR 4-AMINOPHENOL

A-1. DETERMINATION OF INSOLUBLES IN HYDROCHLORIC ACID

A-1.1 Reagents

A-1.1.1 *Concentrated Hydrochloric Acid*

A-1.1.2 *Dilute Hydrochloric Acid* — 5 percent (*m*/*v*).

A-1.2 Procedure — Weigh accurately about 2 g of the sample. Quantitatively transfer to a 250-ml beaker and make a paste with a little water. Add 60 ml of water and 6 ml of concentrated hydrochloric acid. Stir at room temperature till the material is dissolved. (DO NOT HEAT.) Filter through a tared sintered glass G-3 or Gooch crucible. Wash the residue with dilute hydrochloric acid till the washings are colourless or faint amber in colour. Finally, wash with water till free from chlorides. Dry the crucible at 100°C to constant mass. Cool in a desiccator and weigh.

A-1.3 Calculation

$$\text{Insolubles in hydrochloric acid, percent by mass} = \frac{M_1 \times 100}{M}$$

where

M_1 = mass in g of the residue, and

M = mass in g of the material taken for the test.

A-2. DETERMINATION OF 2-AMINOPHENOL CONTENT

A-2.0 Outline of the Method — The estimation of this impurity is done by thin layer chromatography.

A-2.1 Apparatus

A-2.1.1 *Thin Layer Chromatography Plate* — glass plate, 20 × 20 cm, coated uniformly with silica gel powder G (neutral).

A-2.1.2 *Micropipette* — 5 microlitre capacity.

A-2.1.3 *Developing Chamber* — suitable rectangular jar which is closed well with a lid.

A-2.1.4 *Chromatographic Sprayer*

A-2.2 Reagents

A-2.2.1 *Methanol*

A-2.2.2 *2-Aminophenol* — pure (reference sample).

A-2.2.3 *Ehrlich Reagent* — Dissolve 1 g of 4-amino dimethyl benzaldehyde in 25 ml of concentrated hydrochloric acid and 75 ml of methanol.

A-2.2.4 *Eluent* — mixture of benzene, methanol and acetic acid (80 : 14 : 6).

A-2.3 Procedure — Weigh accurately about 2 g of the material and transfer it to a 100-ml volumetric flask and dissolve in methanol. Make up the volume to 100 ml. Similarly weigh accurately about 200 mg of the reference sample and dissolve in 100 ml of methanol (solution A). From solution A, prepare 5 different solutions of 0·2, 0·4, 0·6, 0·8 and 1 percent concentrations. Using a micropipette, spot 5 microlitre each of the sample solution and the reference solutions on the thin layer plate. Care shall be taken to see that all spots are in the same line. Allow the plate to stand for five minutes in the dark so that the spots applied dry up. Place the developer in the chamber. Close the chamber with its lid and allow to achieve equilibrium. Now place the plate carefully in the chamber and allow the mobile phase to run in ascending manner to a distance of about 15 cm from the spots. This will take approximately 1½ hours. Remove the plate from the chamber, dry it in the air and spray with Ehrlich reagent. Warm the plate slightly. After a few minutes examine visually the intensity of colour developed with the material under test and compare it with the known reference samples.

A-2.4 Reporting — Report the 2-aminophenol content as that which is close to the intensity of the standard. In case the colour intensity of the test sample does not come within the range of the standard spots, repeat the experiment using a different percentage of the material.

INDIAN STANDARDS

ON

DYE INTERMEDIATES

IS:
2630-1975 Nitrobenzene (*first revision*)
2740-1973 Sulphanilic acid, technical (*first revision*)
2741-1973 β-Naphthol (*first revision*)
2744-1964 α-Naphthylamine
2833-1973 Aniline, technical (*first revision*)
3229-1973 Naphthionic acid (sodium salt) (*first revision*)
3242-1965 β-Oxy naphthoic acid (BON ACID)
3562-1965 *p*-Nitrotoluene, technical
4265-1975 4, 4′-Diaminostilbene 2, 2′-disulphonic acid (*first revision*)
4334-1967 *o*-Chloroaniline
4335-1967 *m*-Chloroaniline
4336-1967 *p*-Chloroaniline
4425-1967 *p*-Nitrotoluene-*o*-sulphonic acid
4523-1968 Acetoacetanilide
4524-1968 Acetoacet-*o*-chloroanilide
4525-1968 *p*-Aminoacetanilide
4526-1968 2, 5-Dichloroaniline
4527-1968 2-Nitro-4-chlorotoluene
4528-1968 4, 4′-Dinitrostilbene-2, 2′-disulphonic acid (disodium salt)
5042-1969 1-Aminoanthraquinone
5043-1969 2-Aminoanthraquinone
5044-1969 Benzanthrone
5045-1969 Metanilic acid, technical
5299-1969 Methods of sampling and tests for dye intermediates
5438-1969 Nitrobenzene-*m*-sulphonic acid, sodium salt
5646-1970 *p*-Anisidine
5647-1970 *p*-Toluidine
5648-1970 *o*-Anisidine
5649-1970 *o*-Toluidine
6258-1970 *o*-Nitroanisole
6259-1971 Anthraquinone, technical
6260-1971 *p*-Nitroanisole
6264-1971 J-acid
6265-1971 Quinizarine, technical
6266-1971 1, 4-Diaminoanthraquinone, technical
6961-1973 3-Bromobenzanthrone, technical
6962-1973 3, 9-Dibromobenzenthrone, technical
6977-1973 1, 5-Diaminanthraquinone, technical
7359-1974 1-Chloroanthraquinone, technical
7360-1974 1, 5-Dichloroanthraquinone, technical
7362-1974 Tobias acid
7364-1974 *m*-Nitro-*p*-toluidine

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INDIAN STANDARDS

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Chemicals, inorganic (miscellaneous)
Chemicals, organic (miscellaneous)
Coal and coke
Coal carbonization products
Coated fabrics
Cosmetics and toilet goods
Dental materials
Drying oils
Dye intermediates
Electroplating chemicals
Explosive and pyrotechnic materials
Fertilizers
Fillers, stoppers and putties
Footwear
Glass and glassware
Industrial gases
Inks and allied products
Laboratory glassware, thermometers and related apparatus
Lac and lac products
Leather, leather goods and leather dressings
Linters and allied products
Lubricants and related products
Oil pastes
Oils & fats, oleaginous seeds and fruits
Painters' materials (miscellaneous)
Paper and its products
Paper and pulp board packaging materials
Perfumery materials, natural and synthetic
Petroleum and petroleum products
Photographic chemicals
Pigments and extenders
Plastics
Polishes
Printing inks
Ready mixed paints and enamels
Rubber and rubber products
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